

Microfluidic platform for multi-parameter water quality analysis

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Outline

- **Introduction**
- **System design and development**
- **Phosphate sensor design and deployments**
- **Other parameters**
- **Future plans & Conclusions**

Introduction

Motivation

- Monitoring of environmental water quality is predominantly based on manual sampling followed by laboratory analysis
- Expensive → Limited in terms of frequency and number of locations
- Not sufficient to satisfy the demands of environmental legislation (e.g. EU WFD)



Autonomous sensors

- Can provide high-frequency data from a larger number of locations with lower cost

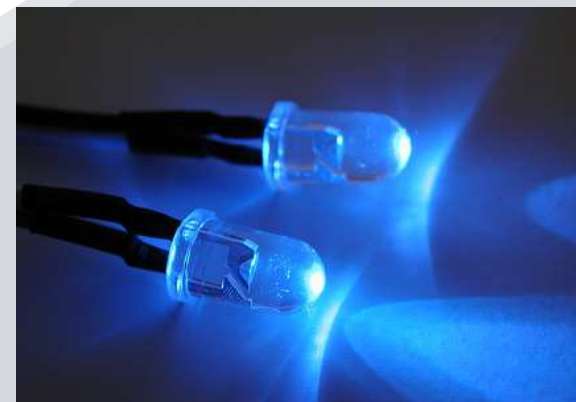
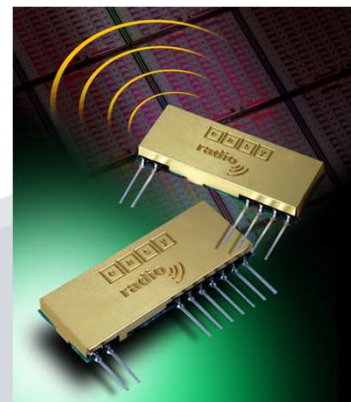
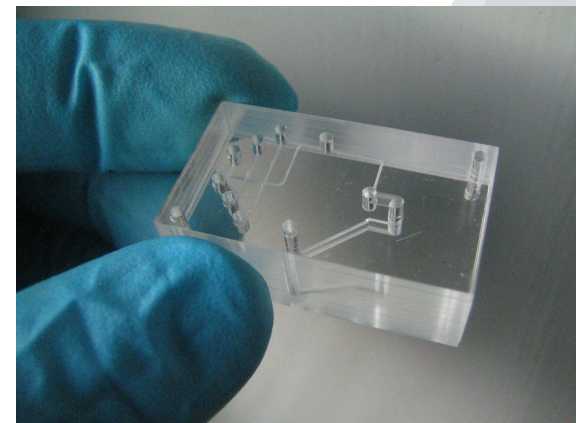


System requirements

- Reliable and sensitive detection system
- Long deployable lifetime ⇔ Low power consumption
- Wireless communication of data to user
- Rugged and portable design
- Low cost, Low maintenance

Approach

- Colorimetric reagent chemistry
- Microfluidic technology
- LED-based detection systems
- Communication by GSM / short range radio (e.g. ZigBee)



Key factors in system design

Chemistry

- Sensitive detection (mg/L - μ g/L)
- Reagent stability
- Simple methods (number of reagents/steps)
→ simple, robust microfluidic chips

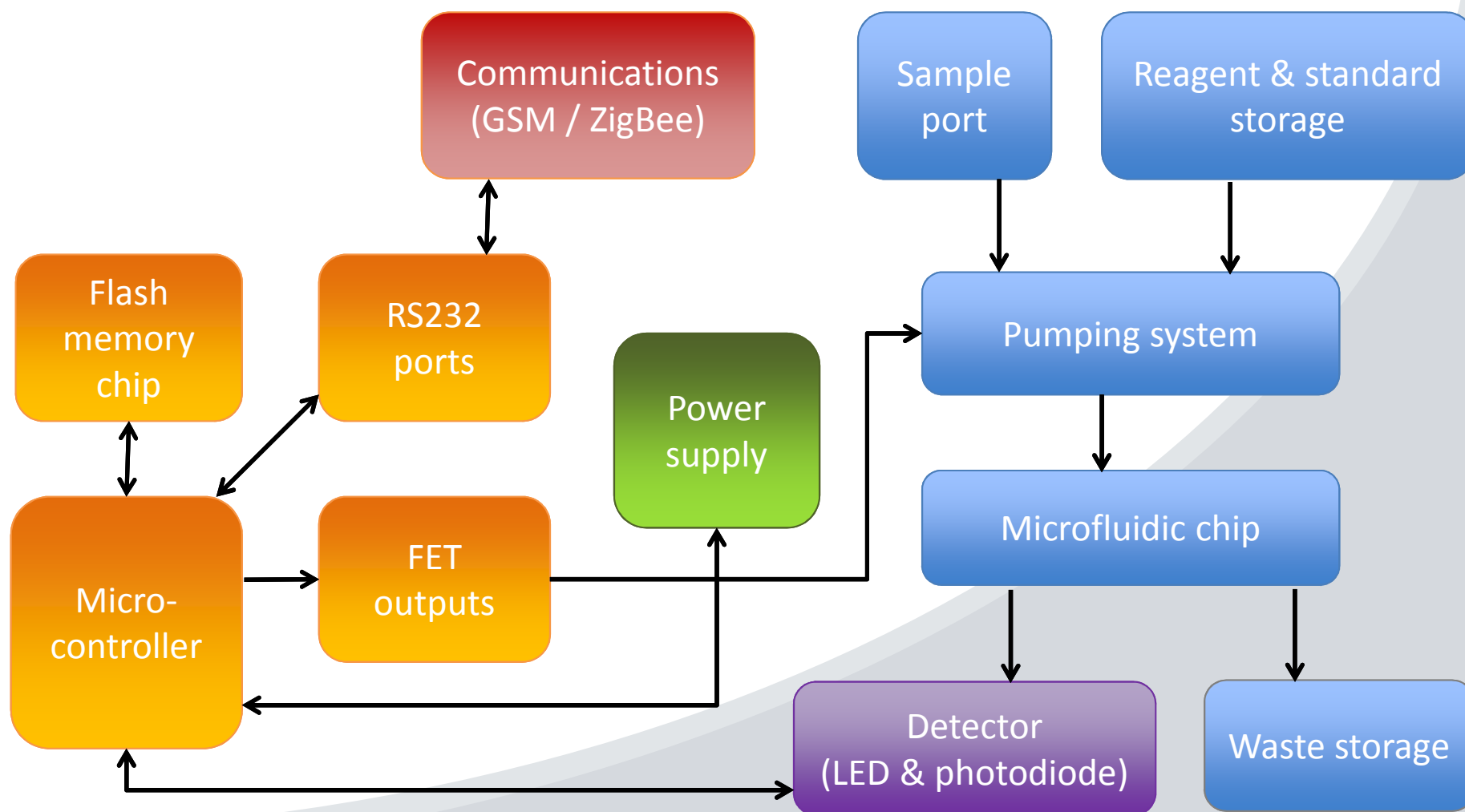
Detectors

- LED & photodiode or Paired Emitter Detector Diodes (PEDDs)
- Sensitive, Low power consumption, Low cost

Communications

- Application dependent (range, frequency, power demands)

System design



Phosphate analyser

3 stages of development

- Demonstration of method (chemistry & detector) in microfluidic manifold
- Development and field testing of prototype system
- Commercialisation (Episensor Ltd., Ireland)

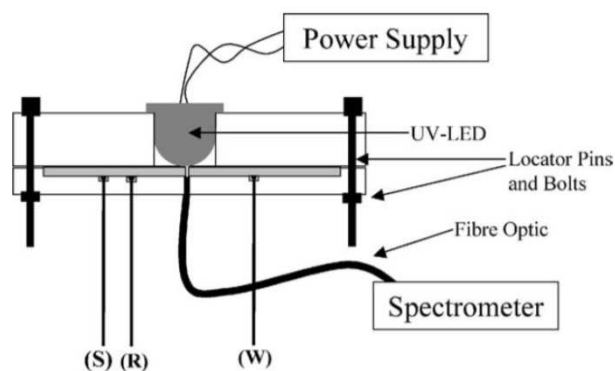
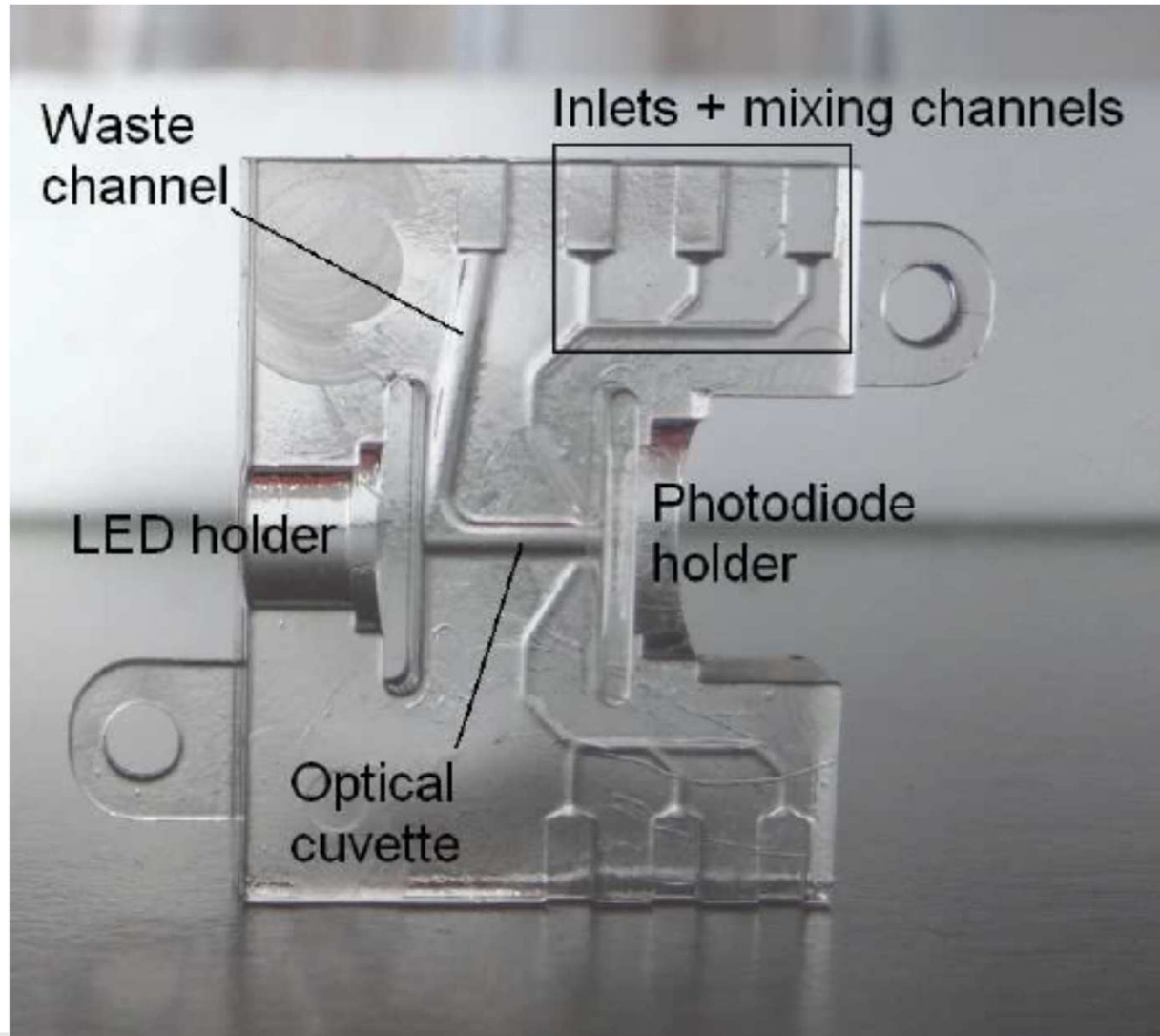


Fig. 1. Cross-section schematic of the optical alignment through the microfluidic chip.



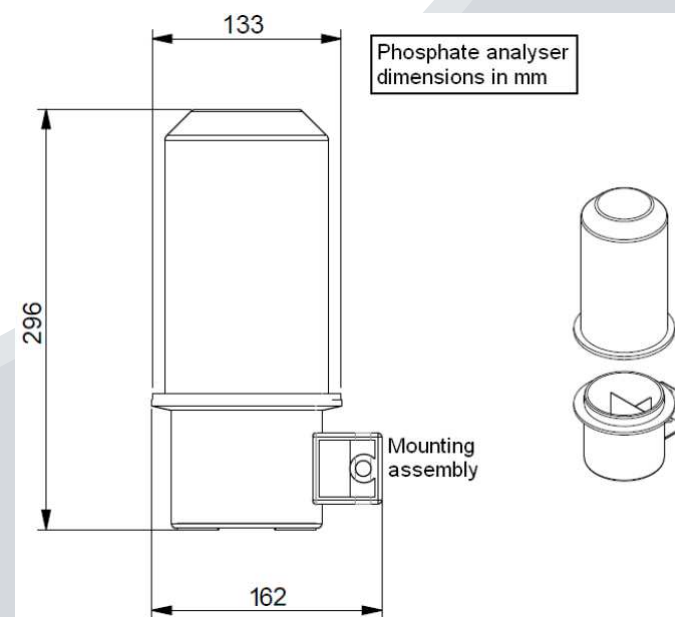
Phosphate sensor



- (1) Sample inlet;
- (2) Control board and detection system;
- (3) Dual channel peristaltic pumps;
- (4) Reagent bags;
- (5) IP68 enclosure

Specifications

- Minimum sample interval: 15 minutes
- Linear response range: 0-20 mg/L PO_4^{3-}
- Limit of detection: 0.20 mg/L PO_4^{3-}
- Up to 1000 assays before reagent/battery replacement
- Weight 1.8 kg with full reagent load

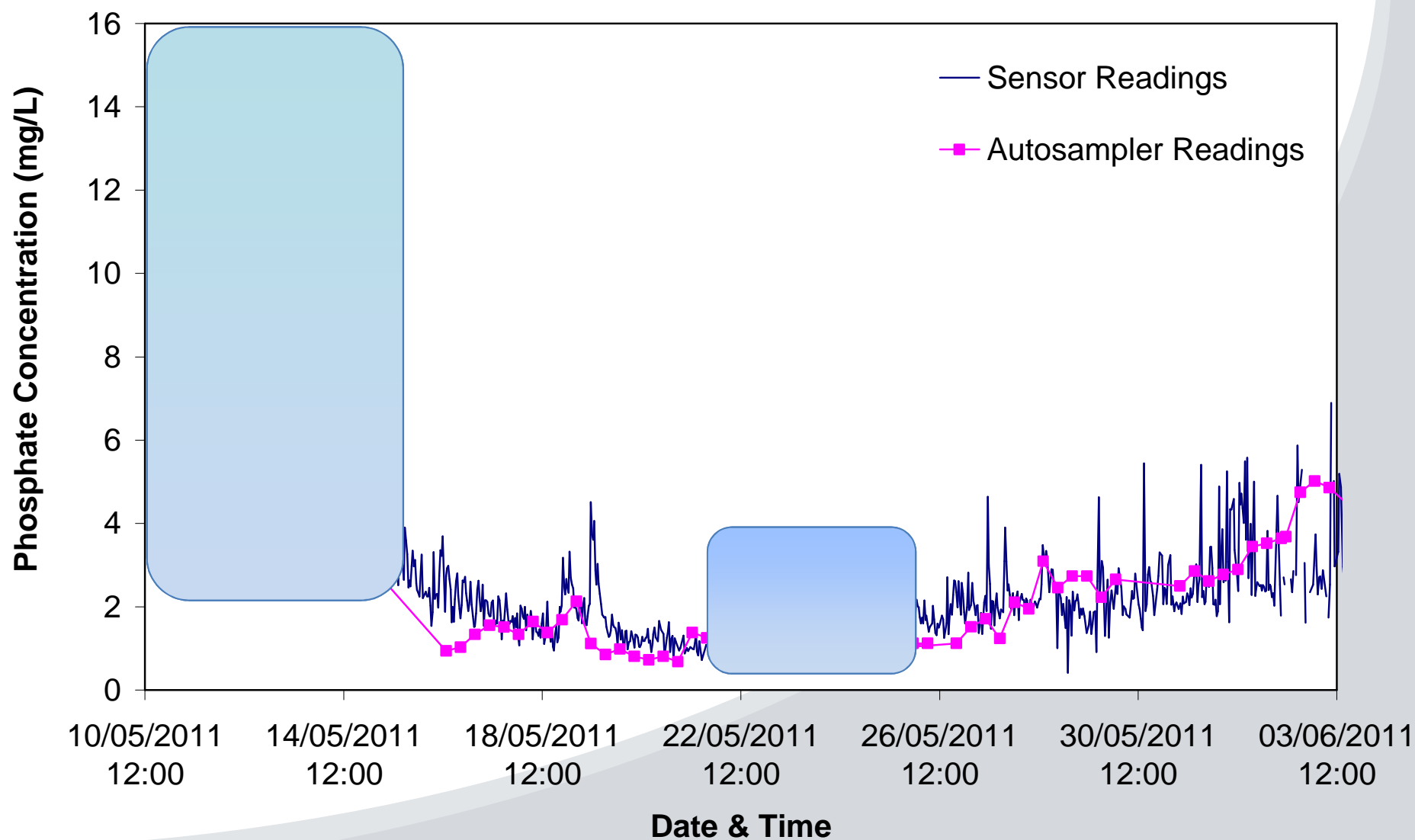


Deployments (i) Wastewater



- WWTP in Co. Kildare, Ireland
- Sensor installed in effluent discharge tank
- 30 min sample interval
- Autosampler collecting 24 samples/week for validation

Deployments (i) Wastewater

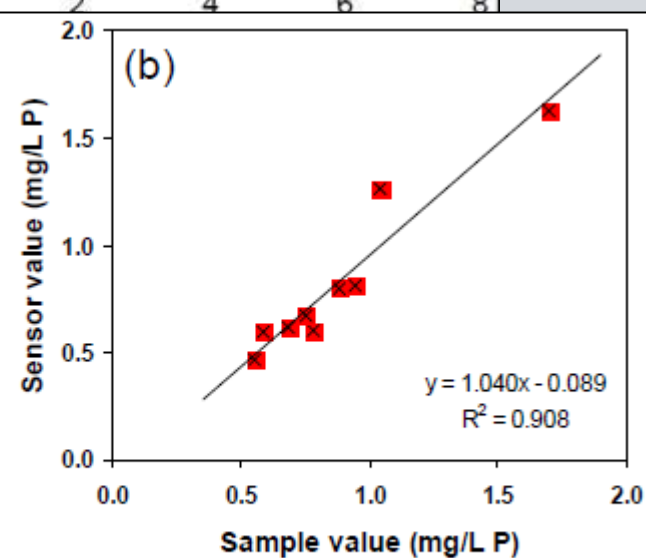
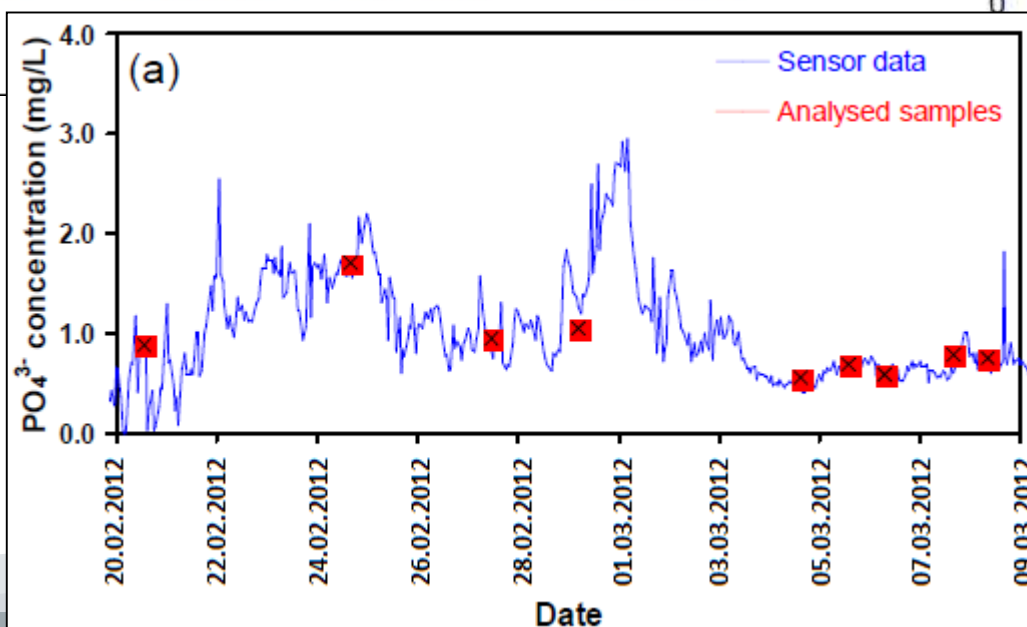
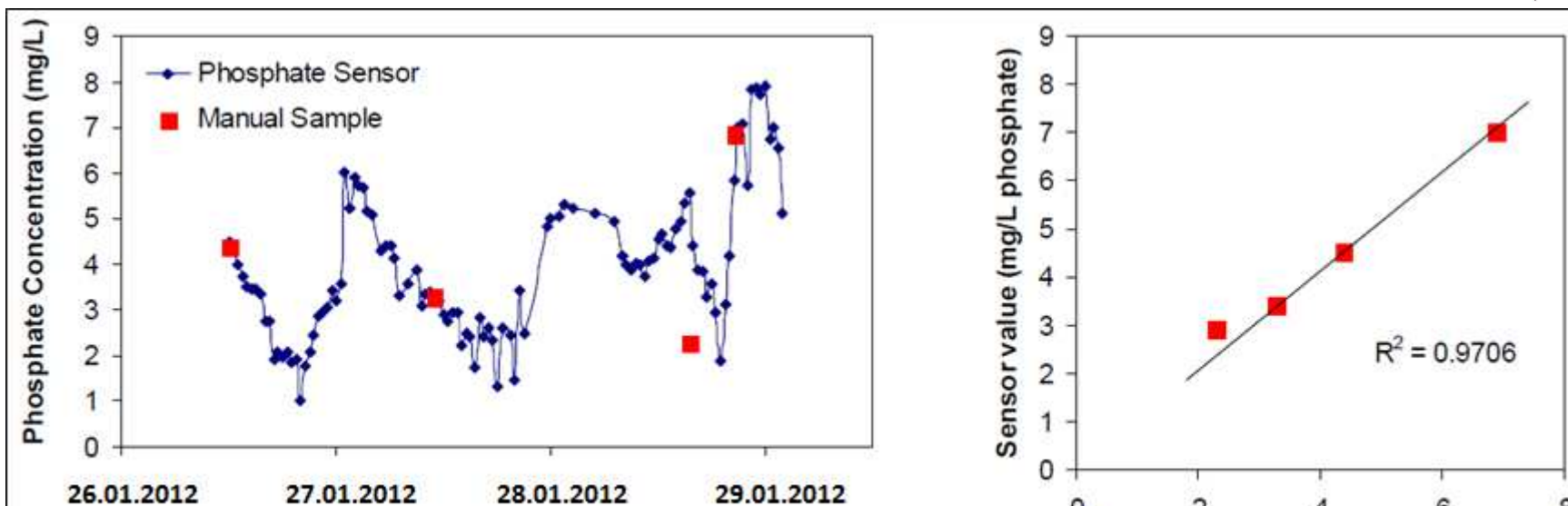


Deployments (ii) Estuary



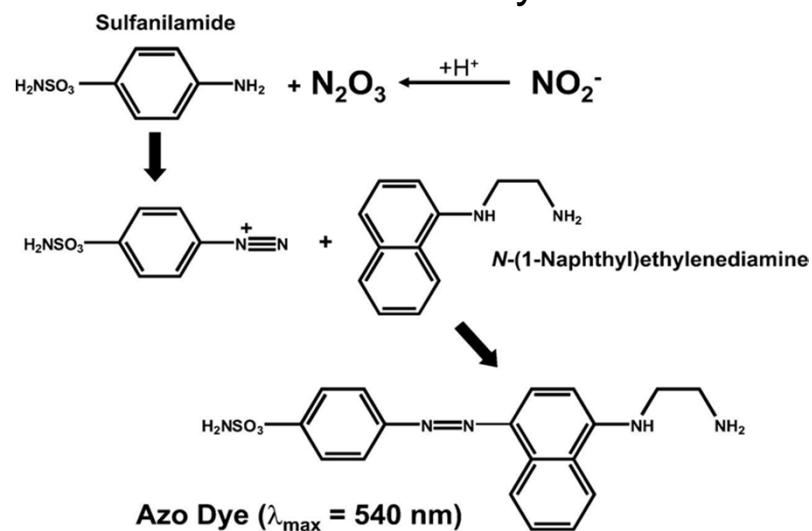
- Broadmeadow Water
- Enclosed estuary north of Dublin City
- Highly impacted - municipal wastewater + agricultural inputs

Deployments (ii) Estuary

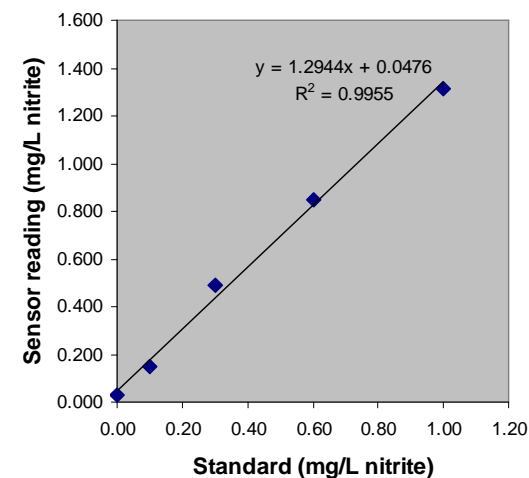


Nitrite detection

Griess assay



- Single reagent → can be incorporated into platform with minimal changes
- Detection at 540 nm
- Detection limit 5 $\mu\text{g/L}$

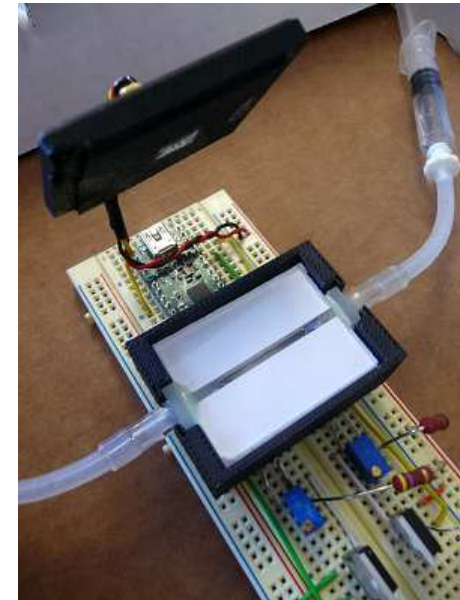
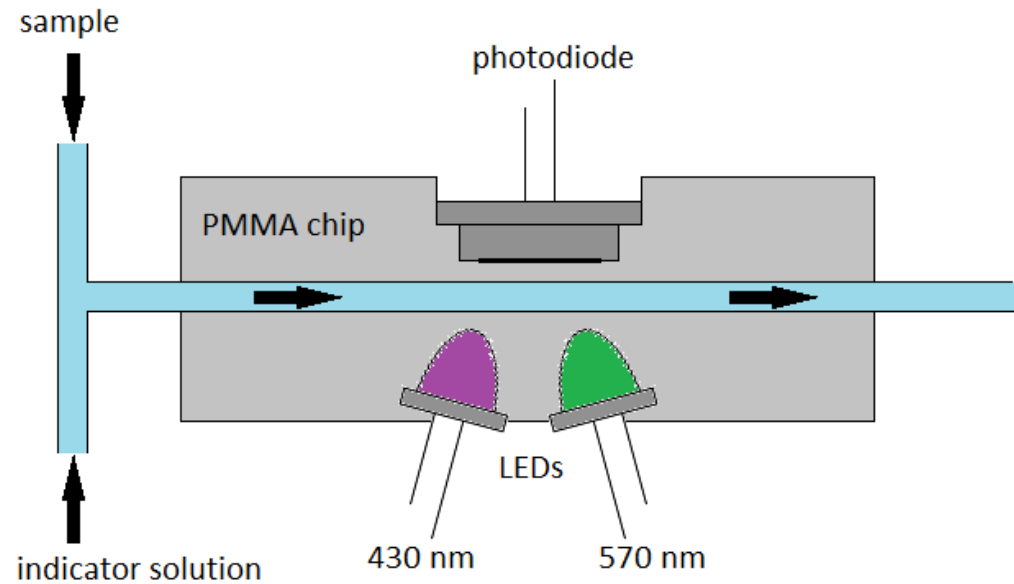


Nitrite detection – blind test

| Sample Reference/Type | Sample ID | Sample Concentration 17/02/2012 by IC | Sample Concentration 21/02/2012 by HACH (LL) | Sample Concentration 21/02/2012 by IC | <u>DCU</u> <u>results</u> <u>22/02/12</u> |
|---|-----------|---|--|---|---|
| 100413 | A | <0.2 | 0.016 | <0.2 | 0.115 |
| 100413 Spiked with 1mg/l NO ₂ | A* | 0.98 | 1.020 | 0.92 | 1.190 |
| 100343 | B | <0.2 | 0.008 | <0.2 | 0.023 |
| 100343 Spiked with 1mg/l NO ₂ | B* | 0.94 | 1.006 | 0.85 | 0.914 |
| 100291 | C | <0.2 | 0.012 | <0.2 | 0.161 |
| 100291 Spiked with 1mg/l NO ₂ | C* | 0.96 | 1.002 | 0.92 | 1.342 |
| 1mg/l NO ₂ standard | D | 0.89 | 0.870 | 0.77 | 1.142 |
| 2mg/l NO ₂ standard | D* | 1.71 | 1.865 | 1.72 | 1.920 |

Samples and external analyses supplied by TE Laboratories Ltd.

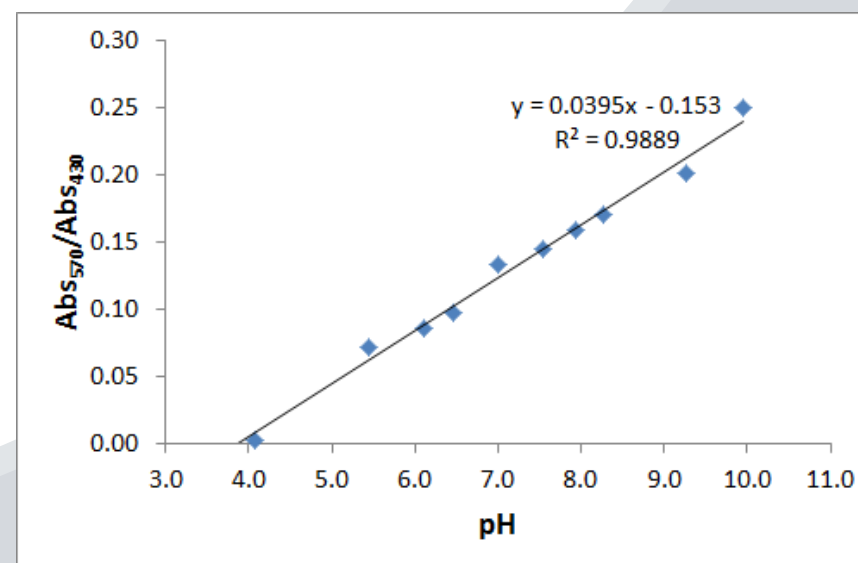
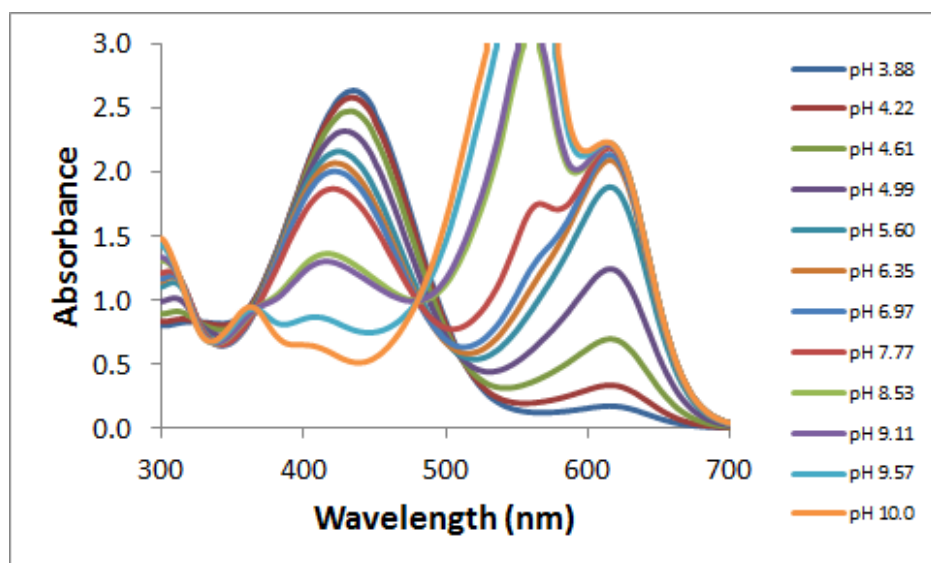
pH measurement



- Mixed indicator solution
- Dual LEDs with adjustable intensity
- Photodiode detector

Mixed indicator solution

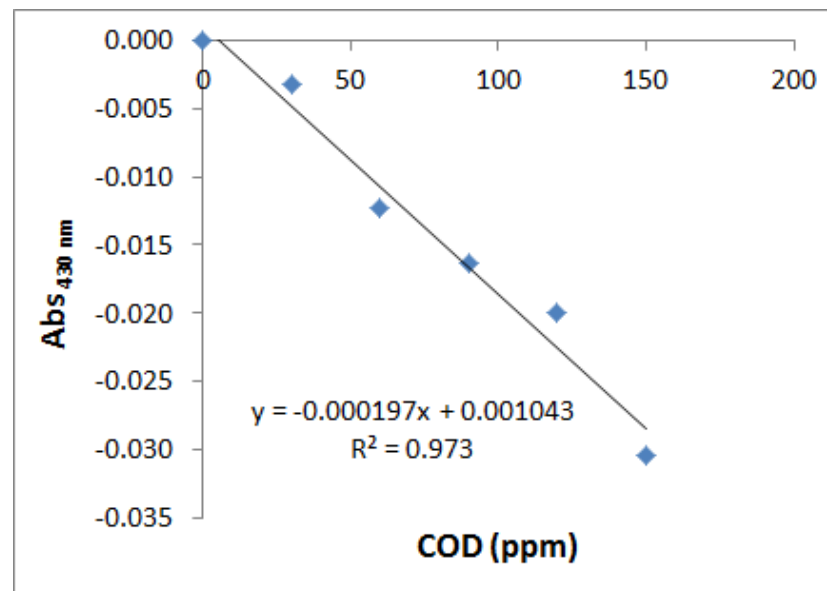
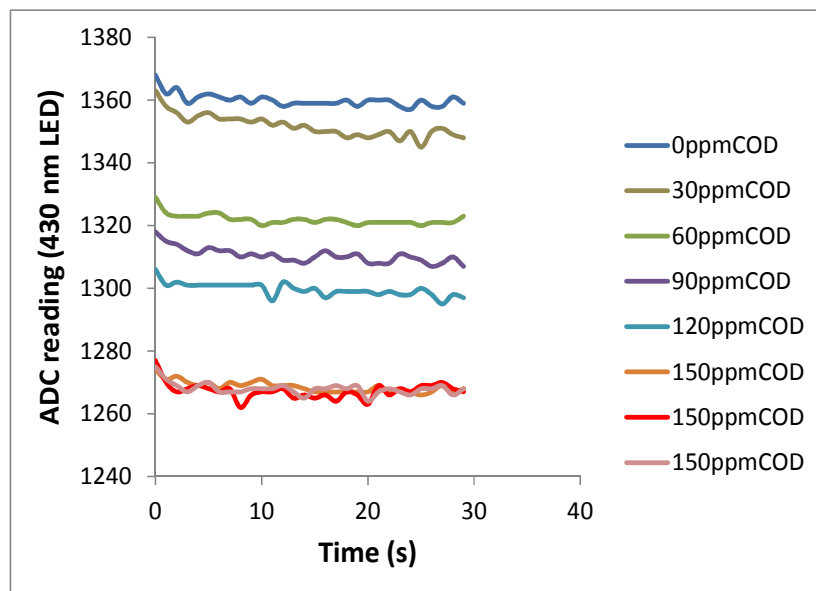
- Colour change over range pH 4 - 10
- 3 options for detection wavelength
- Linear response using ratio of 430 and 570 nm peaks



Chemical Oxygen Demand (COD)

- Standard COD method based on oxidation using potassium dichromate, conc. H_2SO_4 , heating to 150°C for 2 hours
- Colour change from chromate ion ($\text{Cr}_2\text{O}_7^{2-}$, orange) to chromic ion (Cr^{3+} , green)
- Monitor decrease in chromate peak at 430 nm

Detector output

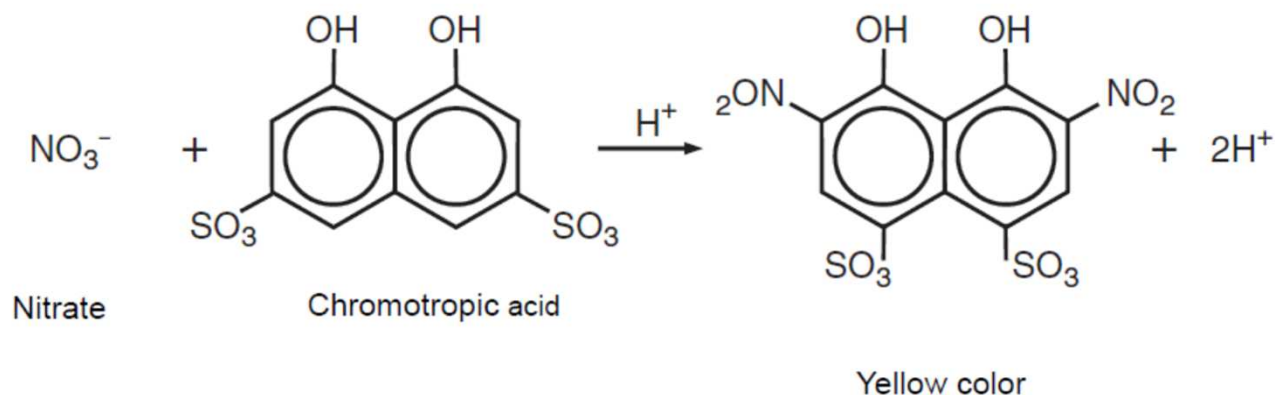


Issues

- Strong acid → resistant materials → more expensive
- Heating → high power consumption

Nitrate detection

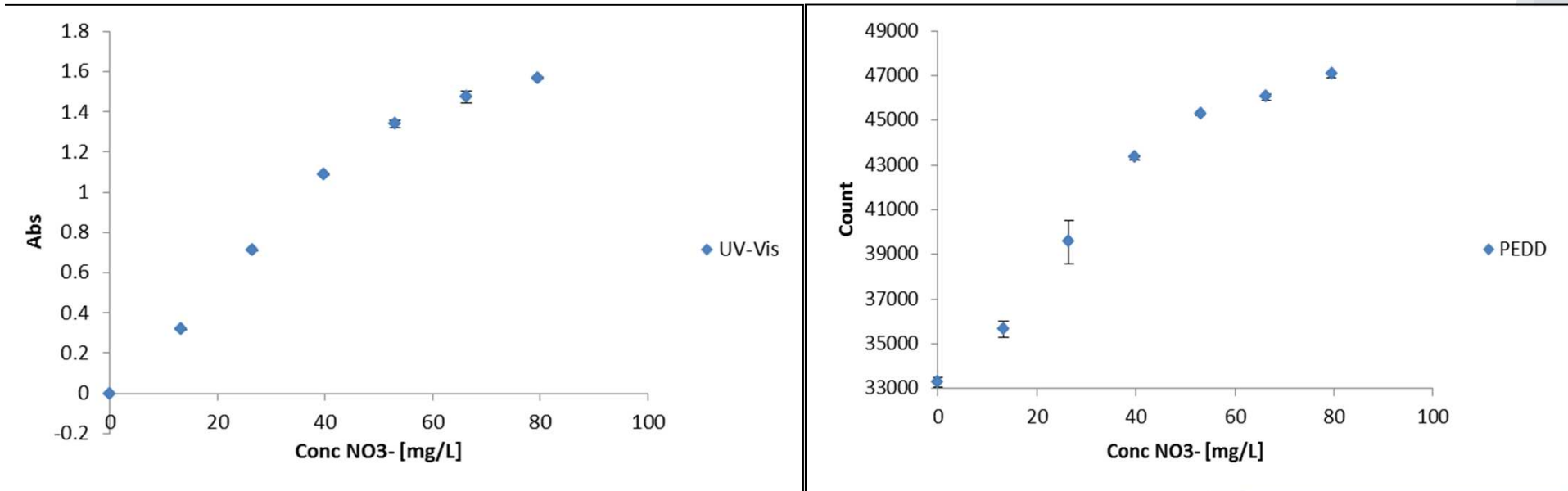
Chromotropic acid method



- Simplified version of literature method* in which reagents (CA + conc. H_2SO_4) added separately with cooling
- Reagents can be combined into single solution, cooling step eliminated

* Ryan, J., Estefan, G. and Rashid, A. 2001. *Soil and Plant Analysis Laboratory Manual*. Second Edition. Syria. ICARDA and NARC 2001.

Calibration

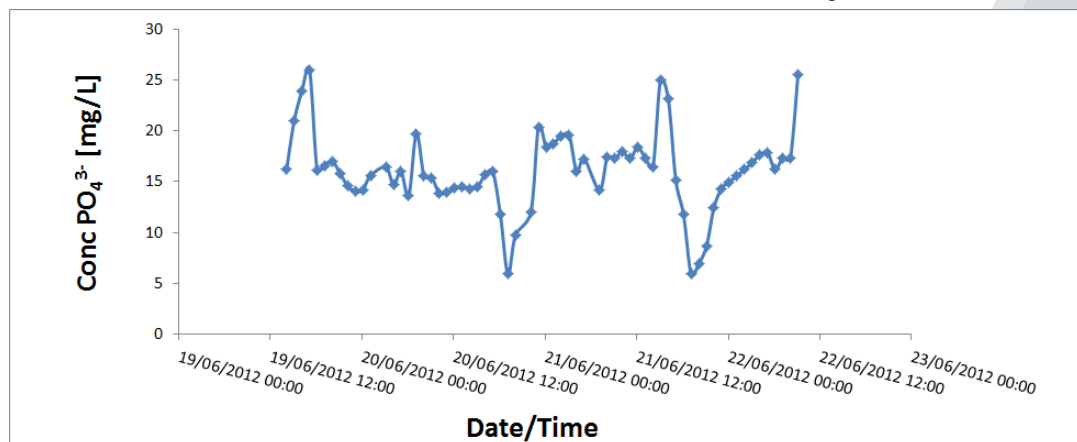


- Calibration curves by UV-vis and LED-based detection
- Linear response to approx . 50 mg/L
- Suitable for environmental and drinking water applications

Future work

Phosphate

- Long term deployment of multiple sensors at sites of interest
- Recently installed at constructed wetlands treatment system



pH, Nitrite

- Field-testing of prototype units



Future work

COD

- Optimisation of detection method in microfluidic system using dual wavelength detection (430 and 600 nm)
- Development of prototype for wastewater monitoring

Nitrate

- Optimisation of chromotropic acid method
- Development of prototype (drinking water)

Conclusions

Deployable sensor for phosphate has been developed and successfully field tested in wastewater, freshwater and estuarine conditions

Prototypes for nitrite and pH have been developed and are ready for field testing

Methods for COD and nitrate are being optimised

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